environments, in which such micas can be expected, are potassic ultrabasic rocks such as kimberlites, mica peridotites, and perhaps mica carbonatites, etc. A search of the literature revealed a number of analyses of phlogopites which show an excess over 3.0 Si in the tetrahedral position. Rimšaite (1964) gives six analyses of such high-silica phlogopites with Si up to 3.18 from various rocks in the Bancroft area, Ontario. The authors are presently searching for further Al-deficient phlogopites.

Preliminary phase relations in the system K_2O-MgO-SiO_2-H_2O indicate that the phase $KMg_{2.5}[Si_4O_{10}](OH)_2$ described in this note is only one end member of a whole series of mica solid solutions in this system. Further researches as to the nature of the lattice substitution leading to this series are under way.

It is also worth noting that attempts at synthesizing an Fe^{2+} analogue of the mica here described have been successful.

REFERENCES

BOWEN, N. L. AND O. F. TUTTLE (1949) The system MgO-SiO_2-H_2O. *Bull. Geol. Soc. Am.* **60,** 439–460.

CROWLEY, M. S. AND R. ROY (1964) Crystalline solubility in the muscovite and phlogopite groups. *Am. Mineral.* **49,** 348–362.

KARDYMOWICZ, I. (1960) Celadonite from Barcza in the Swietokryskie Mountains (Central Poland). *Kwart. Geol.* **4,** 609–616.

MOREY, G. W. (1917) The ternary system H_2O-K_2SiO_3-SiO_2. *Jour. Am. Chem. Soc.* **39,** 1173–1229.

RIMŠAITE, I. (1964) On micas from magmatic and metamorphic rocks. *Beitr. Min. Petr.* **10,** 152–183.

ROEDDER, E. W. (1951) The system K_2O-MgO-SiO_2. *Am. Jour. Sci.* **249,** 81–130, 224–248.

WISE, W. S. AND H. P. EUGSTER (1964) Celadonite: synthesis, thermal stability and occurrence. *Am. Mineral.* **49,** 1031–1083.

YODER, H. S. AND H. P. EUGSTER (1954) Phlogopite synthesis and stability range. *Geochim. Cosmochim. Acta* **6,** 157–185.

THE AMERICAN MINERALOGIST, VOL. 50, JULY-AUGUST, 1965

A METHOD FOR TAKING BACK REFLECTION OSCILLATION PHOTOGRAPHS OF SINGLE CRYSTALS

L. G. BERRY AND S. V. L. N. RAO, *Miller Hall,*
Queen's University, Kingston, Ontario

In *x*-ray diffraction studies "back reflection" geometry is utilized in powder cameras (Cullity, 1956; Azaroff and Buerger, 1958) and in Weissenberg cameras (Buerger, 1937) for the accurate measurement of high

Bragg angles, permitting the determination of lattice parameters with greater precision. Back-reflection Laue photographs are widely used by metallurgists for determining crystal orientation in metal samples which are too large for normal Laue geometry. In the study of epitaxial and topotactic intergrowths it is often necessary to study the surface of crystals larger than those used in the usual single crystal studies. Geometrical relations between exsolved phases or between various sub-ordered units during polymorphic phase transformation, or reactions taking place during corrosion or oxidation and in other allied processes are readily studied using large single crystals and a back reflection oscillation technique.

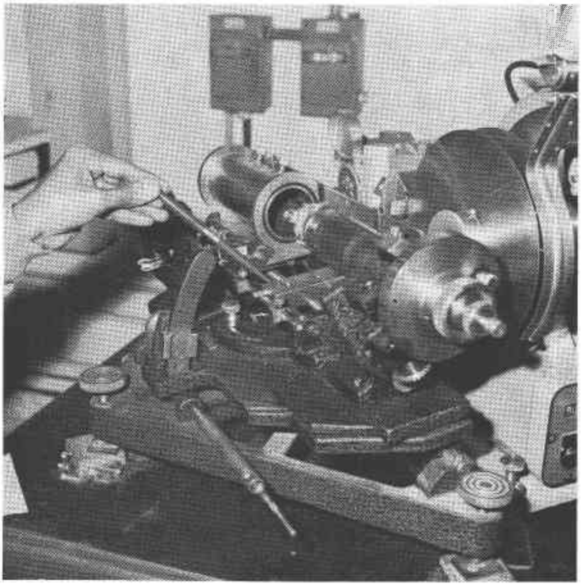

FIG. 1. The Weissenberg unit with the brass block in position. The film unit is moved farther towards the motor end to show the brass block.

Some standard Weissenberg and oscillation cameras are limited to a maximum oscillation angle of about 30°. With this small oscillation angle only a few reflections can be recorded with filtered copper radiation from a cleavage plane of galena of 5 to 15 mm square. The Weissenberg goniometer (an early model by Charles Supper) was readily altered to give an oscillation up to 140° by using an additional brass block. To utilize the instrument (Fig. 1) for large angle oscillation photographs, the entire block carrying the film is moved to the motor end of the lead screw (left hand end), and remains disengaged from the lead screw. The additional brass block with a threaded semicircular slot is mounted on the lead screw ahead of the film unit. This block (Fig. 2) translates as a consequence of rotation of the lead screw (the film unit remains stationary), and the projecting brass pin on the block actuates the motor reversal "stops" on the switching rod at the end of a particular translatory movement of the block. As the rotation of the specimen is coupled to that of the lead screw, the motor reversals cause the oscillation of the specimen. The positions of the "stops" on the switching rod can be varied to obtain the desired oscillation range. The *x*-ray photograph can be recorded in the central part of the mounted film by pushing the film unit towards the beam collimator, after removing a stop screw. The collimator tube has to be shortened so that it does not interfere with the specimen during oscillation. The extent of oscillation should be adjusted so that during the oscillatory movement the *x*-ray beam always strikes on the *same surface* of the crystal at various angles of incidence.

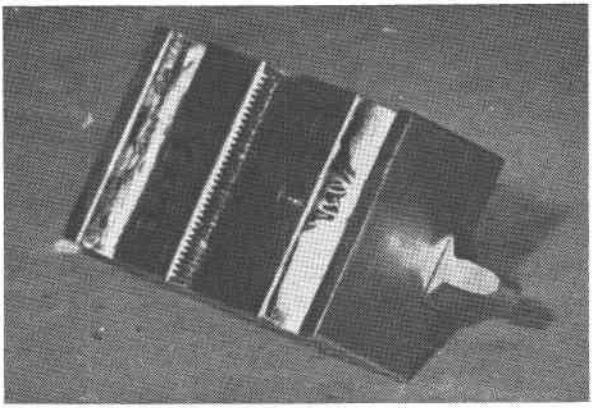

FIG. 2. A closer view of the brass block with the threaded semicircular slot.

Using the above attachment, oscillation of the crystal surface, 60° to either side from the mean position (normal to the crystal surface) is easily possible. The crystal selected for *x*-ray study with a clean large surface is mounted on a standard goniometer head and is aligned using a two circle goniometer. If the axis of oscillation of the crystal does not coincide with the axis of the cylindrical camera, the location of various reflections will be affected. If d spacings are to be deduced from measurement of these films, the specimen surface may be smeared with the powder of a standard substance. The standard substance gives a "powder photograph" and the reflections of the specimen under investigation can be measured with reference to that of the powder lines of the standard substance. In the investigation of oriented transformations during heat treatment, the essential geometrical relations between the various individual components is more important than their lattice dimensions. A study of the oriented transformation of calcite to CaO during heat treatment is to be reported shortly.

ACKNOWLEDGMENTS

One of the authors (Rao) is thankful to the National Research Council of Canada for the award of Postdoctorate fellowship and to the Director, Indian Institute of Technology, Kharagpur (India) for sanctioning him leave to take up this award. He is also thankful to Dr. T. C. Bagchi of the same Institute for the continued encouragement he has given the author in the pursuit of mineralogical studies.

REFERENCES

AZAROFF, L. V. AND M. J. BUERGER (1958) *The Powder Method*, McGraw-Hill Book Co., New York.

BUERGER, M. J. (1937) The precision determination of the linear and angular lattice constants of single crystals. *Zeit Krist.* **97**, 433–468.

CULLITY, B. D. (1956) *Elements of X-ray Diffraction*, Addison-Wesley, Reading, Mass.

THE AMERICAN MINERALOGIST, VOL. 50, JULY–AUGUST, 1965

P-T DEPENDENCE OF THE SPHALERITE⇌WURTZITE INVERSION IN ZnS

A. J. MAJUMDAR AND RUSTUM ROY, *The Pennsylvania State Univ., University Park, Penn.*

In a comprehensive study designed to test the applicability of the Clapeyron relationship to various types of polymorphic transformations, the transition in ZnS has recently been reexamined. The main object in this study was to determine the p-T dependence of the sphalerite⇌wurtzite transition and thereby estimate the heat of transition from the p-T data employing the well known Clapeyron relationship. Kullerud (1953) has used the Clapeyron relationship to predict an increase of 20° C. in the sphalerite⇌wurtzite inversion temperature for every 1000 atmosphere increase in pressure. Precise measurements of unit cell parameters of the two modifications *at room temperature* and a calorimetrically determined value of 3190 cals/mole for the heat of transition obtained from the thermochemical literature provided the known variables in his estimate. However, as recent investigations of Smith (1955) and Strock and Brophy (1955) and others have shown, polymorphism in ZnS is a very com-